

Final Report

Development of Biaxially Texture Coated Superconductors Based on Copper & Copper Alloys

June 1, 2001 – June 30, 2004

**AFOSR 217179/1017809/1
U.S. Air Force Office of Scientific Research**

Program Objectives

The overall program goal was to develop a new class of biaxially textured YBCO conductors using copper or copper-alloys as substrates. To accomplish this goal, the following program tasks were performed: (1) The processing of thin buffer layers on copper based substrates as epitaxial templates and diffusion barriers, and (2) the optimization of buffer-layer structures from the current state-of-art multilayer system to 1-2 layer system by enhancing adhesion between buffer layers and copper based substrates.

Accomplishments

We have developed a method to grow self-aligned epitaxial MgO/Cu/MgO films on silicon substrates by pulsed laser deposition (PLD) technique. Here, a thin layer of Cu/Mg (Mg 5%) is deposited using a PLD over Si (100) specimens, followed by annealing at 500 °C in a controlled oxygen environment resulting in the segregation of Mg on either side of the copper film. Mg on the upper side of copper reacts with ambient oxygen and on the lower side with the adsorbed oxygen in the substrate to form layers of MgO. High-resolution transmission electron microscopy (HRTEM) measurements showed thin layers of MgO formed on either side of the copper films. The lower MgO layer acts as a diffusion barrier and inhibits the diffusion of Cu into the system while the upper MgO layer acts as a passivating layer and protects copper against oxidation. This approach has been used to grow high quality epitaxial $\text{YBa}_2\text{Cu}_3\text{O}_{7-8}$ films with MgO and MgO/TiN acting as a buffer layers for the superconducting device applications.

Thin-film deposition was performed in a multi-target stainless-steel laser deposition chamber evacuated by a turbo-molecular pump to a base pressure of 1×10^{-8} Torr. Radiation from a KrF excimer laser (Lambda Physik 210, $\lambda = 248$ nm) was used for the ablation of TiN and Cu-Mg targets. The stoichiometric hot pressed TiN was obtained from CERAC Inc. and Cu-Mg (5 at. % Mg) was deposited by placing Magnesium chips on copper. These targets were mounted on a rotating multi-target polygon assembly, which were ablated using focused laser beams. The energy density of the beam was $2-3 \text{ Jcm}^{-2}$ at 45° angle of incidence. In this study, epitaxial TiN barrier layers of 60-70 nm thickness were deposited at optimized conditions with substrate temperature of 650°C and a base pressure of 5×10^{-7} Torr. In case of high-temperature superconductor thin film

20050309 080

REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-05-

0070

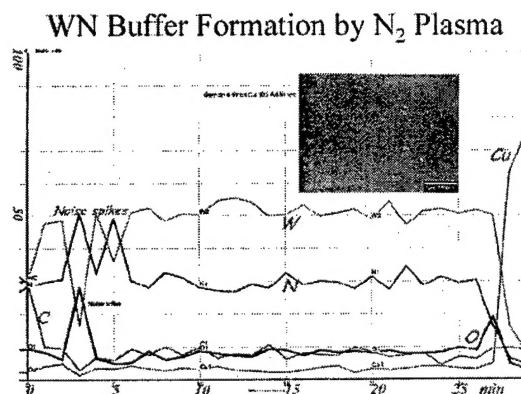
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to the Department of Defense, Executive Service and Communications Directorate, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Project Director (0470-0047), Washington, DC 20503.

PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ORGANIZATION.

1. REPORT DATE (DD-MM-YYYY) 02/18/2005		2. REPORT TYPE Final		3. DATES COVERED (From - To) 01-06-01 to 06/30/04	
4. TITLE AND SUBTITLE Development of Biaxially Textured Coated Superconductors based on Copper and Copper alloys				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER F49620-01-1-0369	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Shaw, David T.				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University at Buffalo, State University of New York Amherst, NY				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Research Foundation of State University of New York 520 Lee Entrance, Suite 211 Amherst, NY 14228 NR				10. SPONSOR/MONITOR'S ACRONYM(S) AFRL/AFOSR	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Distribution unlimited					
13. SUPPLEMENTARY NOTES None					
14. ABSTRACT The primary focus of this project is to grow high-T _c (Y123) films on textured copper substrates using TiN and WN buffer layers. We have developed a method to grow self-aligned epitaxial MgO/Cu/MgO films on silicon substrates by pulsed laser deposition (PLD) technique. A thin layer of Cu/Mg (Mg 5%) is deposited using a PLD over Si and Cu specimens, followed by annealing in a controlled oxygen environment resulting in the segregation of Mg on either side of the copper film. In addition, we have also demonstrated the feasibility of growing WN buffer layer using ion implantation techniques. High-resolution transmission electron microscopy (HRTEM) measurements showed thin layers of nitride films act as a diffusion barrier and inhibits the diffusion of Cu into the system. These approaches have been demonstrated to be useful for growing high quality epitaxial YBa ₂ Cu ₃ O _{7-d} films for Cu- or Cu alloys-based superconducting devices.					
15. SUBJECT TERMS Copper substrates, high temperature superconductors, titanium nitrides, tungsten nitrides, buffer layers					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Shaw, David T.
U	U	U	UU		19b. TELEPHONE NUMBER (Include area code) 716 645 3112 x1218

deposition, the substrate was maintained at a temperature of 650°C. The multilayered structure, thus obtained, was analyzed by X-ray diffraction (XRD) using a Rigaku X-ray diffractometer with Cu K α radiation and a Ni filter. The microstructure characterization of these films was carried out by JEOL-2010F. Electrical resistivity measurements were performed in a closed cycle refrigerator in the temperature range of 12-300 K using a conventional four-point probe technique. We have obtained high quality thin films with critical temperature exceeding 90K.

During the contract period, very pure (99.99 at %) copper tapes with biaxially aligned textures were supplied by Oak Ridge National Laboratory with an in-plane and out-of-plane texture FWHM of 7.9° and 7.6°, respectively. WN buffer layers were processed by a technique based on ion implantation of tungsten ions directly into textured copper based substrates.



The figure shows the formation of WN buffer layers on textured copper substrates. The film was formed by nitridation in a nitrogen plasma of a tungsten ion implanted copper substrate.

A problem in the buffer layer formation for the copper based RABiTS tapes is the poor adhesion. To alleviate this adhesion problem, the formation WN by ion implantation on Cu showed promising results. 20 keV W-ions were implanted into Cu with a dosage p-to $10^{16} - 10^{17}$ ions/cm². After WN film formation by heat annealing, the WN films showed good adhesion to Cu substrates.

Growth of high quality epitaxial MgO, TiN and WN films on copper substrates, which are used as buffer layers to grow high-T_c thin films, has been demonstrated. These films on copper substrates are grown via new paradigm of domain matching epitaxy where integral multiple of lattice planes match across the film-substrate interface. The techniques developed in the program provide an essential framework for growing device quality thin films on copper substrates having small as well as large lattice misfits, and thus opens up a new frontier in practical high-T_c conductors.

Personnel Supported

James Ye (SUNY-Buffalo), J Narayan, A Tiwari and A. Chugh (NCSU)

Publications:

"A novel technique for making self-encapsulated and self-aligned copper films", Amit Chugh, A Tiwari, A Kvit and J Narayan, Materials Science and Engineering B103 (2003) 45-48.

"Self Aligned Passivated copper Interconnects", Talk Presentation at the MRS Spring 2002 meeting, San Fransisco.

Interactions/ transitions

Presentations and participation in all AFOSR MURI Program review meeting in Madison, Wisconsin (2001, 2002, and 2003) and the DOE/AFOSR HTC Wire Workshops in Tampa, Florida (2001,2002, and 2003)

Patent Disclosures

A patent disclosure filed at SUNY/Buffalo by D. Shaw on buffer formation on copper substrate.

Honors/ Awards:

J Narayan received ASM Gold Medal (Highest honor of ASM-International), TMS Fellowship Award, Winner of 2004 Edward Demille Campbell Lecture and Prize